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1. REPORT DATE (DD-MM-YYYY) 02/16/2010		2. REPORT TYPE Technical Report - Briefing Charts			3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE Pure Form of LiBOB Salt and the Purification Process Producing Such Form				5a. CONTRACT NUMBER		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S)				5d. PROJECT NUMBER		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Army Research Laboratory Adelphi MD United States				8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Army Research Laboratory Adelphi MD United States				10. SPONSOR/MONITOR'S ACRONYM(S)		
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION/AVAILABILITY STATEMENT A = Approved For Public Release 12/3/2015 No						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT						
15. SUBJECT TERMS						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON	
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (Include area code)	



U.S. Army Research, Development and Engineering Command

# Pure Form of LiBOB Salt and the Purification Process Producing Such Form



***TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.***

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ARL 09-33

APPROVED FOR PUBLIC RELEASE

February 16, 2011

The invention describes the synthesis and purification of a new lithium salt, bis(oxalato) borate (LiBOB).

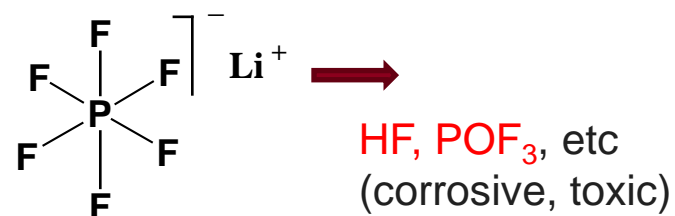
Due to inherent limitations, there is interest in replacing LiPF<sub>6</sub> salt. LiBOB is viewed as a good option because:

- No P-F bond, does not attack organic components
- Does not decompose thermally into HF (as LiPF<sub>6</sub> does); CO<sub>2</sub> as benign products

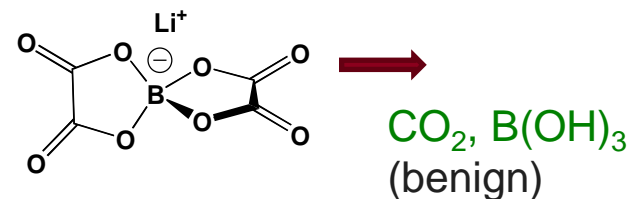
*However, "purified" LiBOB is required to maximize performance benefits. This is currently difficult and expensive to achieve.*

❖ The core technology provided by this invention is the purification procedure, the quality-control standard and the resulting pure form of LiBOB obtained from this process.

❖ This pure form of LiBOB is a distinct compound as compared with other available commercial products.



SOA Electrolyte contains **LiPF<sub>6</sub>**

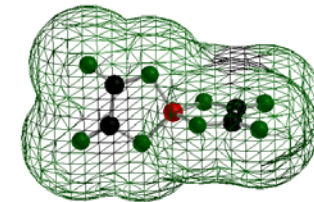


Thermally Stable Electrolyte contains **LiBOB**

## The innovation of preparing pure form of LiBOB and the QC

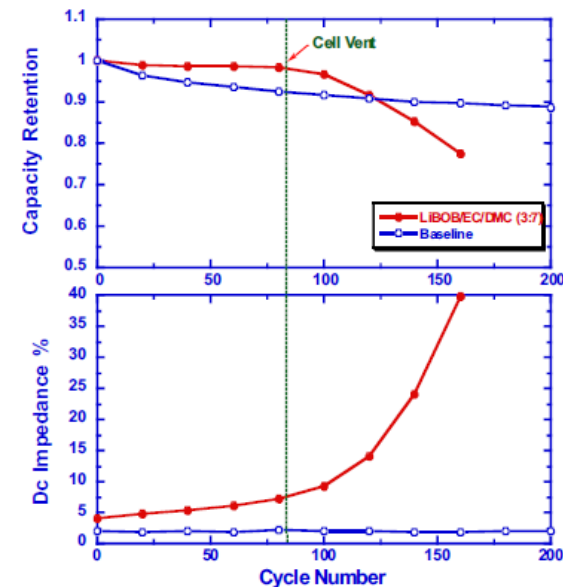
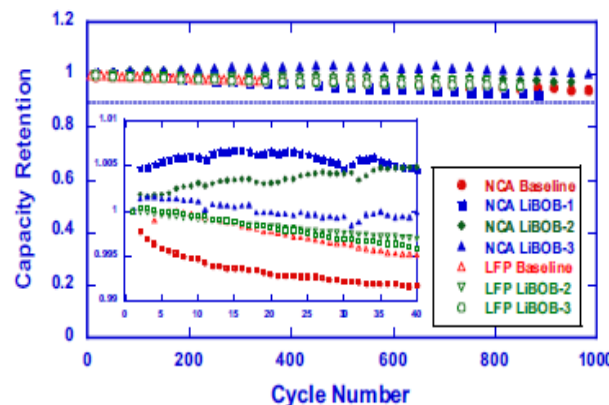
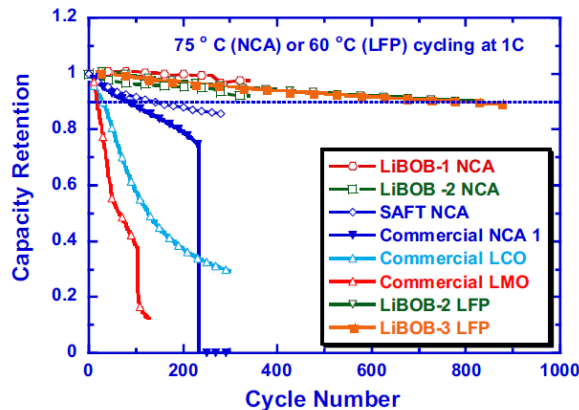
- Impure LiBOB from commercial source cannot support high temperature operation
- The pure form of LiBOB can support Li ion batteries operating at elevated temperatures up to 80 °C
- It also improves safety under abusive over-charge and high-temperature storage

LiBOB



Impure LiBOB does not support HT operation

Pure Form of LiBOB supports HT operation of Industry Li Ion Cells for > 1000 cycles



High temperature stability is critical for battery packs in electrified vehicles

- SOA electrolyte fail to do so
- Become dangerous over 60 °C due to HF production

The pure form LiBOB can widen service temperature range of Li ion batteries

- Dramatically improves capacity retention at both room and high temperature up to 80 °C
- Significantly reduces cell impedance

[Video - SOA Electrolyte \(LiPF<sub>6</sub>\) w/o LiBOB](#)

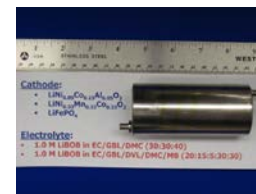
[Video - Electrolyte with pure LiBOB](#)

The invention of the process provides easy production of high purity of LiBOB and its effective Quality Control

Safety advantage over SOA electrolytes

- LiBOB allows large format Li ion cells with higher safety than SOA electrolyte salt LiPF<sub>6</sub>
- Higher stability for both over-charge and HT abuses

8 Ah Li ion cell



Battery Pack in Prius

This invention holds a number of advantages over the current state-of-art:

- Enables the high temperature application of Li ion battery
  - Demonstrates excellent stability at high temperature; up to 80°C
- Is well suited for harsh environments of Hybrid Electric Vehicles (HEV)
- Provides superior performance vs. existing commercial LiBOB; maintains 95% energy density after 1,000 cycles
  - at 75 °C capacity retention ~90% at 1000<sup>th</sup> cycle while most SOA failed before 400<sup>th</sup> cycle
  - at 60 °C capacity improved by 15% vs. SOA at 2000<sup>th</sup> cycle
- Establishes purification process and standard; nearly 100% pure
- Improves safety of Li ion battery under both over-charge and HT abuses
- Open system accommodates a variety of cathode chemistries

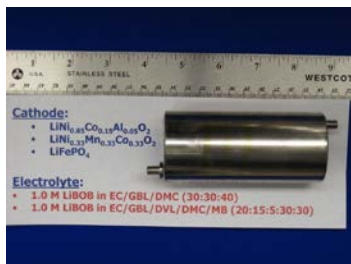
## Method of preparation of these novel additives



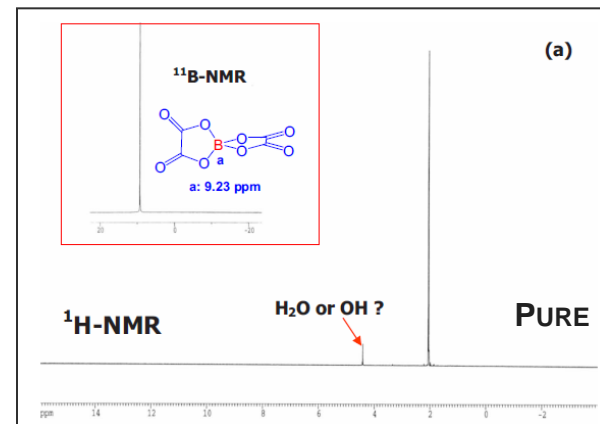
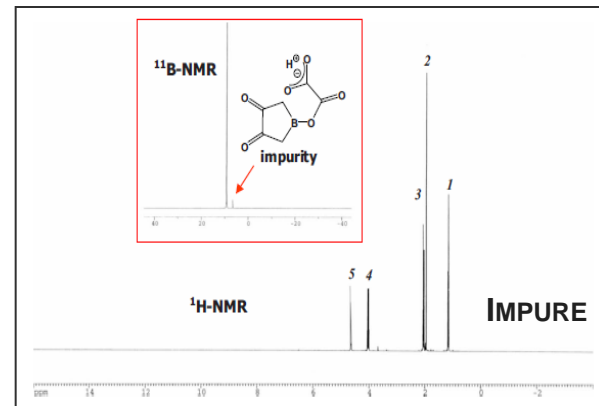
Evaporation/Precipitation  
Recrystallization



## Soxhlet Extraction



Coin Cells  
Industry Cells (8 Ah)



Structural characterization/QC



- Military hybrid electric vehicle applications to reduce fuel consumption and reduce the need for dangerous logistical refueling operations
- Soldier Power in hot climate
  - Battery life significantly improved

**80°C = 176 °F**







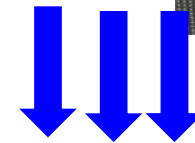
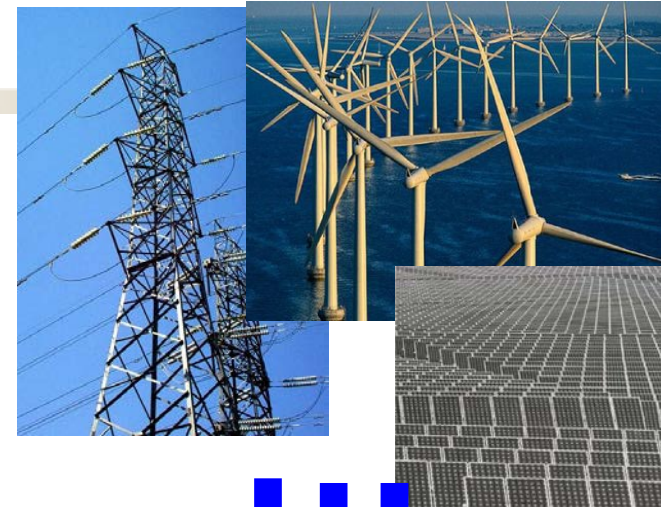
## Two major markets

- Electric Vehicle, Hybrid Electric Vehicle
- Large scale stationary energy storage

The invention provides high temperature stability of Li ion battery.

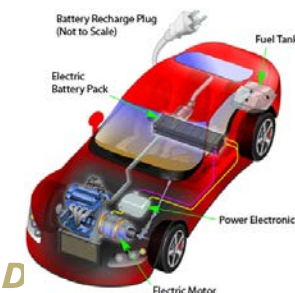
In particular, the invention benefits Li ion battery high temperature applications/environments such as those found in hybrid electric vehicles (HEV).

The purification method developed is also useful for producing other salts that have the BOB anion, such as NaBOB,  $\text{Mg(BOB)}_2$  or other metal salts as additives, ionic liquid for double layer capacitors and batteries, etc.



Energy Storage for Grid Stabilization

Electrified Vehicular Power Systems





# Technology Agreements



A patent license and CRADA is sought.

The current technology is TRL 6 and will benefit from a collaboration between the inventor team and the commercialization partner in order to speed the development to the market. This would most readily be done through a license agreement/CRADA.

A provisional patent application has been filed.